Redetermination of the structure of 4'-(bromomethyl)-[1,1'-biphenyl]-2-carbonitrile, $C_{14}H_{10}BrN$

Maravanahalli S. Siddegowda[I], Hemmige S. Yathirajan[I], Badiadka Narayana[II], Thomas Gerber[III], Eric Hosten[III] and Richard Betz[*, III]

[I] University of Mysore, Department of Studies in Chemistry, Manasagangotri, Mysore 570 006, India
[II] Mangalore University, Department of Studies in Chemistry, Mangalagangotri 574 199, India
[III] Nelson Mandela Metropolitan University, Summerstrand Campus, Department of Chemistry, University Way, Summerstrand, PO Box 77000, Port Elizabeth 6031, South Africa

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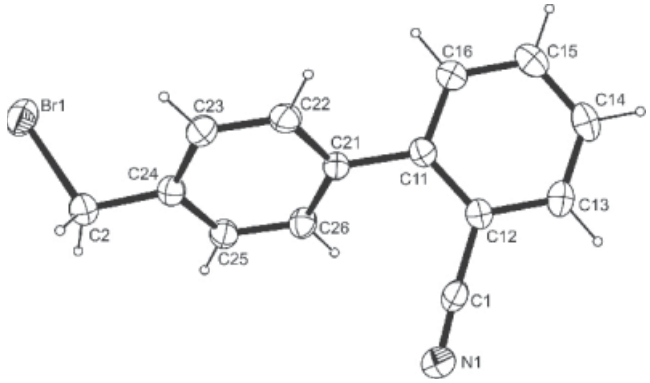

Abstract

$C_{14}H_{10}BrN$, orthorhombic, *Fdd*2 (no. 43), $a = 24.196(1)$ Å, $b = 47.123(2)$ Å, $c = 4.0402(2)$ Å, $V = 4606.5$ Å^3, $Z = 16$, $R_{gt}(F) = 0.0275$, $wR_{ref}(F^2) = 0.0671$, $T = 200$ K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.18×0.309×0.379 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	35.38 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	56.56°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	19485, 2809
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2718
$N(param)_{refined}$:	145
Programs:	SHELX [5], ORTEP-3 [6], MERCURY [7], PLATON [8]

Source of material

The title compound was obtained as a gift sample from R. L. Fine Chem, Bengaluru, India. The compound was recrystallized from acetone by slow evaporation at room temperature.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms, C–H 0.99 Å for the methylene group) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to $1.2U_{eq}$(C).

Discussion

The title compound is used as an intermediate for the synthesis of various biologically active and pharmaceutical compounds. The crystal structure of a related compound, 2-(4-methylphenyl)-benzonitrile, has been reported in the literature [1]. In addition, the crystal structure of the title compound has been determined earlier, however, at room temperature only [2]. In view of the pharmacological importance of the title compound and at the beginning of a more comprehensive study aimed at elucidating the rules guiding structure-activity relationships, the molecular and crystal structure of the title compound was determined at 200 K to allow for comparisons with other members of the series to be synthesized. The title compound constitutes a two-fold-substituted biphenyl derivative bearing a substituent on each aromatic system. The least-squares planes as defined by the carbon atoms of the two individual phenyl rings enclose an angle of 47.36(13)°. The bromine atom is tilted by 59.7(3)° out of the plane of the phenyl ring it is bonded to as part of the bromomethyl substituent. The tilting angle between the two phenyl rings is therefore found to be ~1° smaller than reported for the structure determination at room temperature [2]. In the crystal, C–H···Br as well as C–H···N contacts are observed whose range invariably falls by more than 0.1 Å below the sum of van-der-Waals radii of the respective atoms. Both contacts are exclusively supported by hydrogen atoms on the bromomethyl group. While the C–H···Br contacts connect the molecules to chains along the crystallographic *c* axis, the C–H···N contacts connect the molecules to zig-zag-chains along the crystallographic *a* axis. In total, the molecules form undulated sheets perpendicular to the crystallographic *b* axis. In terms of graph-set analysis [3, 4], the descriptor for the C–H···Br contacts is $C^1_1(3)$ while the descriptor for the C–H···N contacts is $C^1_1(10)$. There is only one intercentroid distance for both bipyridine rings to the rings of adjacent molecules which was measured at 4.0400(15) Å (lattice constant *c*).

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2A)	16*b*	0.1132	0.0561	0.0992	0.036
H(2B)	16*b*	0.1059	0.0729	0.4409	0.036
H(13)	16*b*	0.4640	0.1114	0.3152	0.042
H(14)	16*b*	0.5148	0.0729	0.5127	0.052
H(15)	16*b*	0.4702	0.0308	0.6558	0.050
H(16)	16*b*	0.3748	0.0270	0.6083	0.041
H(22)	16*b*	0.2996	0.0253	0.1826	0.034
H(23)	16*b*	0.2044	0.0203	0.1443	0.034
H(25)	16*b*	0.1843	0.0957	0.6108	0.032
H(26)	16*b*	0.2797	0.1014	0.6329	0.031

* Correspondence author (e-mail: richard.betz@webmail.co.za)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	16*b*	0.09183(1)	0.021650(5)	0.54245(9)	0.0311(1)	0.0349(1)	0.0434(1)	–0.0071(1)	0.0049(1)	–0.0052(1)
N(1)	16*b*	0.33732(9)	0.13504(5)	0.0995(7)	0.034(1)	0.030(1)	0.058(2)	–0.0003(8)	0.004(1)	0.011(1)
C(1)	16*b*	0.3587(1)	0.11585(5)	0.2129(6)	0.024(1)	0.026(1)	0.034(1)	–0.0063(9)	0.0026(9)	0.0014(9)
C(2)	16*b*	0.1228(1)	0.05595(5)	0.3374(7)	0.025(1)	0.029(1)	0.035(2)	–0.0002(8)	–0.005(1)	–0.000(1)
C(11)	16*b*	0.3604(1)	0.06697(5)	0.4383(6)	0.021(1)	0.026(1)	0.028(1)	0.0012(8)	0.0015(8)	–0.0004(8)
C(12)	16*b*	0.38829(9)	0.09200(5)	0.3487(6)	0.021(1)	0.027(1)	0.030(1)	–0.0004(8)	–0.0008(9)	0.0005(9)
C(13)	16*b*	0.4458(1)	0.09427(6)	0.3759(7)	0.023(1)	0.037(1)	0.045(2)	–0.0063(9)	–0.001(1)	0.003(1)
C(14)	16*b*	0.4758(1)	0.07147(6)	0.4918(9)	0.020(1)	0.048(2)	0.061(2)	0.002(1)	–0.005(1)	0.004(2)
C(15)	16*b*	0.4493(1)	0.04652(6)	0.5776(9)	0.029(1)	0.040(1)	0.056(2)	0.008(1)	–0.006(1)	0.009(1)
C(16)	16*b*	0.3924(1)	0.04432(5)	0.5499(9)	0.027(1)	0.029(1)	0.045(1)	0.0023(9)	–0.002(1)	0.007(1)
C(21)	16*b*	0.29948(9)	0.06395(5)	0.4138(6)	0.021(1)	0.024(1)	0.027(1)	–0.0012(8)	–0.0012(8)	0.0031(8)
C(22)	16*b*	0.27615(9)	0.03973(4)	0.2676(7)	0.028(1)	0.0224(9)	0.034(1)	0.0028(8)	0.005(1)	–0.002(1)
C(23)	16*b*	0.2196(1)	0.03671(5)	0.2461(7)	0.029(1)	0.024(1)	0.034(1)	–0.0026(8)	–0.002(1)	–0.003(1)
C(24)	16*b*	0.18440(9)	0.05766(5)	0.3734(5)	0.024(1)	0.027(1)	0.022(1)	–0.0012(8)	–0.0008(8)	–0.0002(8)
C(25)	16*b*	0.20765(9)	0.08144(4)	0.5202(7)	0.024(1)	0.0240(9)	0.033(1)	0.0009(8)	0.001(1)	–0.003(1)
C(26)	16*b*	0.26453(9)	0.08475(4)	0.5366(7)	0.026(1)	0.0255(9)	0.028(1)	–0.0026(8)	0.001(1)	–0.005(1)

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